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10/813,697	03/31/2004	Jean Pol Dodelet	2465 US	6137

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George A. Seaby  
Seaby & ASSOCIATES  
603 - 880 Wellington Street  
Ottawa, ON K1R 6K7  
CANADA

EXAMINER

MARKHAM, WESLEY D

ART UNIT	PAPER NUMBER
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1762

DATE MAILED: 01/11/2005

Please find below and/or attached an Office communication concerning this application or proceeding.

## Office Action Summary

**Application No.**

10/813,697

**Applicant(s)**

DODELET ET AL.

**Examiner**

Wesley D Markham

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-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

### Period for Reply

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If the period for reply specified above is less than thirty (30) days, a reply within the statutory minimum of thirty (30) days will be considered timely.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

### Status

- 1) ☐ Responsive to communication(s) filed on \_\_\_\_.
- 2a) ☐ This action is **FINAL**. 2b) ☒ This action is non-final.
- 3) ☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

### Disposition of Claims

- 4) ☒ Claim(s) 1-21 is/are pending in the application.
- 4a) Of the above claim(s) \_\_\_\_ is/are withdrawn from consideration.
- 5) ☒ Claim(s) 16,17,19 and 21 is/are allowed.
- 6) ☒ Claim(s) 1-10 and 12 is/are rejected.
- 7) ☒ Claim(s) 11,13-15,18 and 20 is/are objected to.
- 8) ☐ Claim(s) \_\_\_\_ are subject to restriction and/or election requirement.

### Application Papers

- 9) ☒ The specification is objected to by the Examiner.
- 10) ☒ The drawing(s) filed on 31 March 2004 and 22 July 2004 is/are: a) ☒ accepted or b) ☐ objected to by the Examiner.

Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).

Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).

- 11) ☐ The oath or declaration is objected to by the Examiner. Note the attached Office Action or form PTO-152.

### Priority under 35 U.S.C. § 119

- 12) ☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).
- a) ☐ All b) ☐ Some \* c) ☐ None of:
- ☐ Certified copies of the priority documents have been received.
  - ☐ Certified copies of the priority documents have been received in Application No. \_\_\_\_.
  - ☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

\* See the attached detailed Office action for a list of the certified copies not received.

### Attachment(s)

- |   |   |
|---|---|
| 1) <input checked="" type="checkbox"/> Notice of References Cited (PTO-892)             | 4) <input type="checkbox"/> Interview Summary (PTO-413)                     |
| 2) <input type="checkbox"/> Notice of Draftsperson's Patent Drawing Review (PTO-948)    | Paper No(s)/Mail Date. ____.  |
| 3) <input type="checkbox"/> Information Disclosure Statement(s) (PTO-1449 or PTO/SB/08) | 5) <input type="checkbox"/> Notice of Informal Patent Application (PTO-152) |
| Paper No(s)/Mail Date ____.   | 6) <input type="checkbox"/> Other: ____.                                    |

### **DETAILED ACTION**

1. **Claims 1 – 21** are currently pending in U.S. Application Serial No. 10/813,697, and an Office Action on the merits follows.

### ***Drawings***

2. The formal drawings (3 sheets) filed by the applicant on 3/31/2004 and 7/22/2004 are acknowledged and approved by the examiner.

### ***Specification***

3. The specification is objected to as failing to provide proper antecedent basis for the claimed subject matter. See 37 CFR 1.75(d)(1) and MPEP § 608.01(o). Correction of the following is required: The specification lacks antecedent basis for the following claim limitations: (1) treating the substrate carrying carbon nanotubes (CNTs) with 70% nitric acid for 2 to 20 hours (Claim 5); (2) a silane solution having a platinum and/or ruthenium concentration of 0.02 to 2 M (Claim 12); and (3) reducing the composite at a temperature of 550 to 600° C in a hydrogen-argon atmosphere (Claim 14).

### ***Claim Objections***

4. Claims 8 and 20 are objected to because of the following informalities:
  - Claim 8: The phrase, “the metallic catalyst of is at least one of platinum and ruthenium” appears to contain a typographical error (i.e., the word “of”

between “catalyst” and “is” is unnecessary and confusing), and the applicant is suggested to amend the aforementioned phrase to read, “the metallic catalyst is at least one of platinum and ruthenium”.

- Claim 20: This claim appears to depend from Claim 18 (see the preamble of the claim). However, Claim 20 requires the silane solution to contain both platinum and ruthenium salts, while Claim 18 recites only a ruthenium salt. As such, the preamble of Claim 20 appears to contain a typographical error (i.e., the phrase “claim 18” should read “claim 19” so that the platinum and ruthenium salts recited in Claim 20 have proper antecedent basis).

Appropriate correction is required.

### ***Claim Rejections - 35 USC § 112***

5. The following is a quotation of the second paragraph of 35 U.S.C. 112:

The specification shall conclude with one or more claims particularly pointing out and distinctly claiming the subject matter which the applicant regards as his invention.

6. Claim 12 is rejected under 35 U.S.C. 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention.
7. **Claim 12** requires that “the silane solution contains sufficient to give a concentration of at least one of platinum and ruthenium of 0.02 to 2M”. This limitation renders the scope of Claim 12 vague and indefinite because it is unclear what component is sufficiently contained in the silane solution to give the claimed Pt and/or Ru

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concentration (i.e., the solution contains sufficient what to give the desired concentration?)

8. The following is a quotation of the first paragraph of 35 U.S.C. 112:

The specification shall contain a written description of the invention, and of the manner and process of making and using it, in such full, clear, concise, and exact terms as to enable any person skilled in the art to which it pertains, or with which it is most nearly connected, to make and use the same and shall set forth the best mode contemplated by the inventor of carrying out his invention.

9. **Claims 1 – 10** are rejected under 35 U.S.C. 112, first paragraph, because the specification, while being enabling for performing the claimed process when the silane component in the silane solution is 2(4-chlorosulfonylphenyl) ethyl trichlorosilane, does not reasonably provide enablement for performing the claimed process using a “silane solution” in general, as recited in Claims 1 – 10. The specification does not enable any person skilled in the art to which it pertains, or with which it is most nearly connected, to use the invention commensurate in scope with these claims. Specifically, Claims 1 – 10 are drawn to a process of producing CNTs with metal catalyst particles thereon by immersing a CNT-carrying substrate into a generic “silane solution of a metal catalyst”. After carefully reviewing the applicant’s specification, the examiner notes that the only silane solution taught or disclosed by the applicant is 2(4-chlorosulfonylphenyl) ethyl trichlorosilane. Further, it appears that the mechanism by which the claimed process works is highly dependent on the specific silane compound used / disclosed by the applicant (i.e., 2(4-chlorosulfonylphenyl) ethyl trichlorosilane – see page 4, lines 4 – 13, of the

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applicant's specification, in which it is clearly shown that the chemical nature of the silane compound used in the process is responsible for metal catalyst particle deposition). There is no guidance that would teach one skilled in the art what other silane compounds could be successfully used in the claimed process, and what other silane compounds could not be successfully used. Therefore, the broad genus of a "silane solution" could not have been used in the applicant's claimed process without undue experimentation, and the claims are not enabled as required by 35 U.S.C. 112, first paragraph. Please note that Claims 11 – 21 have not been rejected under 35 U.S.C. 112, first paragraph, because these claims require that the silane compound be 2(4-chlorosulfonylphenyl) ethyl trichlorosilane.

***Claim Rejections - 35 USC § 103***

10. The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all

obviousness rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negated by the manner in which the invention was made.

11. This application currently names joint inventors. In considering patentability of the claims under 35 U.S.C. 103(a), the examiner presumes that the subject matter of the various claims was commonly owned at the time any inventions covered therein were made absent any evidence to the contrary. Applicant is advised of the obligation under 37 CFR 1.56 to point out the inventor and invention dates of each claim that was not commonly owned at the time a later invention was made in order

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for the examiner to consider the applicability of 35 U.S.C. 103(c) and potential 35 U.S.C. 102(e), (f) or (g) prior art under 35 U.S.C. 103(a).

12. Claims 1, 2, 4, 6, and 7 are rejected under 35 U.S.C. 103(a) as being unpatentable over Wang et al. (USPN 6,824,689) in view of Sun et al. ("Growth of carbon nanotubes on carbon paper by Ohmically heating silane-dispersed catalytic sites", 2002).
13. Regarding independent **Claim 1**, Wang et al. teaches a process for producing CNTs with metal catalyst particles thereon, the method comprising the steps of (1) preparing a solution of a metal catalyst (e.g., a cobalt-based Fischer-Tropsch catalyst), (2) immersing a substrate made of a material such as a metal or carbon (i.e., an "electrically conducting substrate"), the substrate carrying CNTs thereon, into the solution to yield a composite structure of the substrate, CNTs, and metallic catalyst, and (3) heating (i.e., reducing) the composite structure to yield a composite of substrate, CNTs, and metallic catalyst particles (Abstract, Col.1, lines 18 – 21 and 46 – 55, Col.2, lines 8 – 15 and 42 – 46, Col.5, lines 40 – 51, Col.6, lines 9 – 21 and 43 – 56, Col.8, lines 10 – 14, Col.9, lines 15 – 67, Col.10, lines 1 – 24, and Col.11, lines 50 – 67, Col.12, and Col.13, lines 1 – 34 (for the deposition of the CNTs on the substrate), and Col.13, lines 35 – 67, and Col.14, lines 1 – 35 (for the deposition and reduction of the catalyst onto the CNT-coated support)). Wang et al. does not explicitly teach that the solution of the metal catalyst is a silane solution. However, the metal catalyst deposited onto the CNTs in the process of Wang et al. is, for

example, a Co catalyst formed by dipping the substrate into a cobalt salt-containing solution (Col.6, lines 53 – 55, and Col.14, lines 25 – 35). Sun et al. teaches that, in the art of depositing cobalt catalyst particles onto a carbon paper support by dip coating (i.e., a process analogous to that of Wang et al.), it is desirable to utilize a solution comprising a silane (i.e., 2(4-chlorosulfonylphenyl) ethyl trichlorosilane) and the metal (i.e., cobalt) catalyst salt because such a silane solution gives a homogeneous distribution of catalytic transition metal ions on the support, thereby giving a better distribution of catalytic particles once the catalytic material is reduced (Abstract, pages 415, 416, and the “Conclusions” section on page 420). Therefore, it would have been obvious to one of ordinary skill in the art to utilize a silane solution of a cobalt catalyst to deposit the catalyst onto the CNTs in the process of Wang et al. with the reasonable expectation of (1) success, as such a silane solution can be successfully used to deposit a metal catalyst onto a carbon-based support (see Sun et al.), and (2) obtaining the benefits of using the silane solution, such as providing a better distribution of the catalyst on the support (as taught by Sun et al.). Regarding **Claims 2 and 4**, Wang et al. also teaches treating the CNT-carrying substrate with nitric acid (Col.10, lines 7 – 15, Col.12, lines 58 – 67). Regarding **Claim 6**, the combination of Wang et al. and Sun et al. also teaches that the catalyst is, for example, cobalt (see the discussion of Claim 1 above). Regarding **Claim 7**, the substrate can be carbon paper (Col.5, line 50 of Wang et al.; the entire Sun et al. document), and the CNTs have a diameter of up to 100 nm (Col.6, line 15 of Wang et al.). Carbon nanotubes having such a large diameter (around 100 nm) are



necessarily multi-walled CNTs, as claimed by the applicant (see, for example, paragraph [0042] of Grigorian et al. (US 2002/0172767 A1) and/or paragraph [0009] of Tour et al. (US 2004/0180244 A1), which are simply cited to show that CNTs having such a large diameter are MWNTs, not SWNTs).

14. Claim 3 is rejected under 35 U.S.C. 103(a) as being unpatentable over Wang et al. (USPN 6,824,689) in view of Sun et al. ("Growth of carbon nanotubes on carbon paper by Ohmically heating silane-dispersed catalytic sites", 2002), in further view of Smalley et al. (USPN 6,752,977).
15. The combination of Wang et al. and Sun et al. teaches all the limitations of **Claim 3** as set forth above in paragraph 13, except for treating the CNT-carrying substrate with methanol. However, Smalley et al. teaches that, after oxidizing CNTs (i.e., a process taught by Wang et al. – Col.10, lines 10 – 15), it is desirable to wash the CNTs in methanol. Therefore, it would have been obvious to one of ordinary skill in the art to wash the oxidized, supported CNTs of Wang et al. in a solvent such as methanol, as taught by Smalley et al., in order to minimize the amount of impurities and contaminants present in the CNTs prior to the metal catalyst deposition.
16. Claim 5 is rejected under 35 U.S.C. 103(a) as being unpatentable over Wang et al. (USPN 6,824,689) in view of Sun et al. ("Growth of carbon nanotubes on carbon paper by Ohmically heating silane-dispersed catalytic sites", 2002), in further view of Niu et al. (US 2003/0086858 A1).

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17. The combination of Wang et al. and Sun et al. teaches all the limitations of **Claim 5**

as set forth above in paragraph 13, except for treating the CNT-carrying substrate with 70% nitric acid for 2 to 20 hours. However, Wang et al. does teach treating the CNT-carrying substrate with nitric acid in general in order to oxidize the nanotubes, thereby making the CNTs more receptive to the subsequently deposited metal catalyst (Col.10, lines 3 – 15, Col.12, lines 66 – 67). Wang et al. is silent regarding the specific nitric acid concentration and treatment time. Niu et al. teaches that nanotubes can be oxidized by treatment in 70% nitric acid for 7 hours (paragraph [0178]). Therefore, it would have been obvious to one of ordinary skill in the art to utilize 70% nitric acid and a treatment time of 7 hours (e.g., 2 to 20 hours, as claimed by the applicant) in the process of Wang et al. because such nitric acid treatment conditions are known to successfully oxidize nanotubes, which is the goal of the nitric acid treatment of Wang et al.

***Allowable Subject Matter***

18. Claims 16, 17, 19, and 21 are allowed. Claims 11, 13 – 15, 18, and 20 are objected to as being dependent upon a rejected base claim, but would be allowable if rewritten in independent form including all of the limitations of the base claim and any intervening claims. Claim 12 has been rejected under 35 U.S.C. 112, second paragraph, but no art has been applied against the claim.

19. The following is a statement of reasons for the indication of allowable subject matter:

The prior art of record, alone or in combination, does not teach or reasonably

suggest performing the applicant's claimed process of producing CNTs with metal catalyst particles thereon, wherein the metal catalyst is at least one of platinum and ruthenium, and the silane solution in which the CNT-carrying substrate is dipped contains a platinum and/or ruthenium salt (e.g., chloride) and 2(4-chlorosulfonylphenyl) ethyl trichlorosilane.

### ***Conclusion***

The prior art made of record and not relied upon is considered pertinent to applicant's disclosure. Toops (US 2004/0224217 A1), Choi et al. (US 2004/0018416 A1), Suenaga et al. (US 2002/0177032 A1), and Steigerwalt et al. ("Pt-Ru/Carbon Fiber Nanocomposites...", 2002) all teach carbon nanotube / nanofiber-based electrodes used in fuel cells, the electrodes containing metal (e.g., Pt, Ru, or Pt-Ru) catalyst particles deposited thereon. However, the catalytic particles are not deposited in the manner claimed by the applicant. Dodelet et al. (US 2003/0202930 A1) teaches a method comprising dipping carbon paper into a solution comprising (1) 2(4-chlorosulfonylphenyl) ethyl trichlorosilane and (2) Co and Ni salts, and then growing CNTs by CVD on the Co/Ni catalyst-coated carbon paper. Easton et al. ("Chemical Modification of Proton Exchange Membrane Fuel Cell Catalysts with a Sulfonated Silane", 2001) and Easton ("Chemical Modification of Fuel Cell Catalysts and Electrochemistry of Proton Exchange Membrane Fuel Cell Electrodes", 2003) teach depositing a sulfonated silane such as 2(4-chlorosulfonylphenyl) ethyl trichlorosilane onto a carbon support (e.g., carbon black) prior to or after depositing a metallic (e.g., Pt)

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catalyst on the carbon support. However, the carbon support of Easton is not nanotube-based, and the silane solution does not comprise Pt and/or Ru salts (e.g., because the Pt or Ru is deposited separately from the sulfonated silane).

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Wesley D Markham whose telephone number is (571) 272-1422. The examiner can normally be reached on Monday - Friday, 8:00 AM to 4:30 PM.

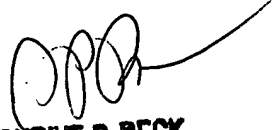
If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Shrive Beck can be reached on (571) 272-1415. The fax phone number for the organization where this application or proceeding is assigned is 703-872-9306.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see <http://pair-direct.uspto.gov>. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free).



WDM

Wesley D Markham  
Examiner  
Art Unit 1762



**SHRIVE P. BECK**  
**SUPERVISORY PATENT EXAMINER**  
**TECHNOLOGY CENTER 1700**